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Mazdoor Kisan Shakti Sangathan

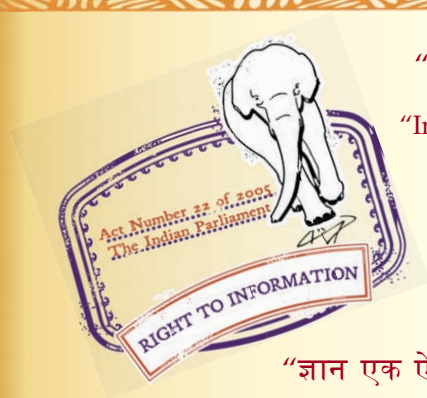
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“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 3547 (1976): Mango Nectar [FAD 10: Processed Fruits and Vegetable Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



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IS : 3547 - 1976

*Indian Standard*  
SPECIFICATION FOR MANGO NECTAR  
( *First Revision* )

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BUREAU OF INDIAN STANDARDS  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

Gr 4

March 1977

# Indian Standard

## SPECIFICATION FOR MANGO NECTAR

### (First Revision)

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**AMENDMENT NO. 1 MAY 1996**  
**TO**  
**IS 3547 : 1976 SPECIFICATION FOR MANGO NECTAR**  
**( First Revision )**

( Page 3, clause 0.5 ) — Insert the following new clause after 0.5 and renumber the subsequent clause:

**'0.6** A scheme for labelling environment friendly products known as ECO-Mark has been introduced at the instance of the Ministry of Environment and Forests (MEF), Government of India. The ECO-Mark shall be administered by the Bureau of Indian Standards (BIS) under the *BIS Act*, 1986 as per the Resolution No. 71 dated 20 February 1991 and Resolution No. 425 dated 28 October 1992 published in the Gazette of the Government of India. For a product to be eligible for marking with the ECO-Mark it shall also carry the Standard Mark of BIS for quality besides meeting additional environment friendly (EF) requirements. The environment friendly requirements for mango nectar are, therefore, included through Amendment No. 1 to this standard.

This amendment is based on the Gazette Notification No. 624 (E) dated 6 September, 1995 for Labelling Beverages, Infant Foods, Processed Fruits and Vegetable Products as environment friendly, published in the Gazette of the Government of India.'

( Page 6, clause 4.8 ) — Insert the following new matter after 4.8:

**"4.9 Additional Requirements for ECO-Mark**

**4.9.1 General Requirements**

**4.9.1.1** The product shall conform to the requirements prescribed under 4.1 to 4.8.

**4.9.1.2** The manufacturer shall produce the consent clearance as per the provisions of *Water (PCP) Act*, 1974, *Water (PCP) Cess Act*, 1977 and *Air (PCP) Act*, 1981 along with the authorization if required under *Environment (Protection) Act*, 1986 and the Rules made thereunder to the Bureau of Indian Standards while applying for the ECO-Mark and the product shall also be in accordance with the *Prevention of Food Adulteration Act*, 1954 and the Rules made thereunder. Additionally, FPO 1955 (Fruit Product Order) framed under *Essential Commodities Act*, 1955, *Standards of Weights and Measures Act*, 1977 requirements wherever applicable has to be complied with.

## **Amend No. 1 to IS 3547 : 1976**

**4.9.1.3** The product/packaging may also display in brief the criteria based on which the product has been labelled environment friendly.

**4.9.1.4** The material used for product/packing shall be recyclable or biodegradable.

**4.9.1.5** The date of manufacture and date of expiry shall be declared on the product/package by the manufacturer.

**4.9.1.6** The product shall be microbiologically safe when tested as per IS 5403 : 1969 'Method for yeast and mould count of foodstuffs' and IS 5887 ( Part 5 ) : 1976 'Methods for detection of bacteria responsible for food poisoning : Part 5 Isolation, identification and enumeration of *Vibrio Cholerae* and *Vibrio Parahaemolyticus* ( first revision )' and shall be free from bacterial and fungal toxins.

**4.9.1.7** The pesticide residues, if any in the product shall not exceed the limit as prescribed in PFA Act, 1954 and the Rules made thereunder.

**4.9.1.8** The product/package or leaflet accompanying it may display instructions of proper use, storage and transport (including refrigeration temperature compliance) so as to maximize the product performance, safety and minimize wastage.

### **4.9.2 Specific Requirements**

**4.9.2.1** The product shall not contain any of the heavy metal contaminants in excess of the quantities prescribed in Table 2."

( Page 8, clause 5.3 ) — Insert the following new clause after 5.3:

### **'5.4 ECO-Mark**

The product may also be marked with the ECO-Mark, the details of which may be obtained from the Bureau of Indian Standards.

( FAD 10 )

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Reprography Unit, BIS, New Delhi, India

***Indian Standard***  
**SPECIFICATION FOR MANGO NECTAR**  
***(First Revision)***

**0. FOREWORD**

**0.1** This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 30 September 1976, after the draft finalized by the Fruits and Vegetables Sectional Committee had been approved by the Agricultural and Food Products Division Council.

**0.2** Mango nectar occupies an important place among the processed fruits manufactured within the country. A sizeable amount of this product is exported. However, there is a considerable scope for further development of this product both for internal as well as export trade. It is, therefore, necessary to ensure the quality of the product which may be done on the basis of this standard.

**0.3** In the preparation of mango nectar, the mango pulp is diluted with sugar syrup so that the pulp content in the nectar is not less than 25 percent by mass.

**0.4** This standard was first issued in 1966. The Sectional Committee responsible for preparation of this standard felt that this specification should be reviewed in the light of latest trade practices. In this revision reference to IS : 6542-1972\* has been made regarding hygienic conditions. Further requirements for acidity has been specified; specific gravity has been specified in degrees brix, and requirements for vacuum, head space and metallic contaminants have been modified. Requirements for packing have also been enlarged.

**0.5** In the preparation of this standard, due consideration has been given to provisions of the Prevention of Food Adulteration Rules, 1955 and the Fruit Products Order, 1955. However, this standard is subject to the restrictions imposed under these statutes, wherever applicable.

**0.6** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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\*Code for hygienic conditions for fruit and vegetable canning units.

†Rules for rounding off numerical values (*revised*).

**IS : 3547 - 1976**

## **1. SCOPE**

**1.1** This standard prescribes the requirements and the methods of sampling and test for mango nectar.

## **2. TERMINOLOGY**

**2.0** For the purpose of this standard, the following definitions shall apply.

**2.1 Head Space** — The distance between the top of the double seam and the level of the surface of the contents.

**2.2 Absence of Defects** — Freedom from extraneous materials, such as peel and fibrous tissue.

## **3. GRADES**

**3.1** Mango nectar shall be of two grades, namely:

Grade 1, and  
Grade 2.

## **4. REQUIREMENTS**

**4.1** Mango nectar shall be prepared from fresh pulp or pulp preserved without any chemicals. The mangoes used shall be free from damage caused by insects, disease, etc, and shall be free from any signs of fermentation. The substances that should be added to the pulp are, water, cane or beet sugar, and citric acid or malic acid or ascorbic acid.

**4.1.1** Any commercially cultivated variety suitable for mango nectar manufacture may be used.

**4.2 Hygienic Requirements** — The material shall be prepared and handled under strict hygienic conditions (see IS : 6542 - 1972\*) by persons free from contagious and infectious diseases. The premises shall be maintained in a thoroughly clean and hygienic condition and shall have adequate and safe water supply. All workers shall wear clean, white, washed clothing. Necessary precautions shall be taken to prevent incidental contamination of the product from soiled equipment or from personnel suffering from injuries.

**4.2.1** All equipment coming in contact with raw materials or products in the course of manufacture shall be kept clean. An ample supply of steam and water and hose, brushes and other equipment necessary for proper cleaning of machinery and equipment, shall be available. The equipment shall be properly cleaned with suitable chlorine solution having 50 mg/kg of available chlorine.

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\*Code for hygienic conditions for fruit and vegetable canning units.

**4.3 Colouring Matter, Flavouring and Synthetic Sweetening Agents** — The material shall be free from artificial colouring matter and flavouring, and synthetic sweetening agents. However, it may contain  $\beta$ -carotene.

**4.4 Fruit Content** — The mango pulp content in the nectar shall be not less than 25 percent by mass on as-is basis.

**4.5** The ethanol content of mango nectar shall not exceed 3 g/kg when tested according to the method given in the 'Indian Standard method of test for determination of ethanol (*under preparation*)'.

NOTE — Till such time the standard under preparation is published, the method of test shall be as agreed to between the concerned parties.

**4.6 Finished Product** — The contents of the can on opening shall display the following characteristics:

*Grade 1* — The mango nectar shall possess a good body; shall be of a uniform colour; shall possess a normal characteristic taste and flavour; shall be practically free from defects; and shall score not less than 85 points.

*Grade 2* — The mango nectar shall possess a good body; shall be of reasonably uniform colour; shall possess a normal characteristic taste and flavour; shall be reasonably free from defects; and shall score not less than 75 points.

The maximum and minimum number of points to be scored by different factors shall be as follows. Scoring shall be done according to the method prescribed in Appendix A.

Factor	Maximum	Minimum	
		Grade 1	Grade 2
Colour	25	19	16
Taste and flavour	50	37	33
Absence of defects	25	19	16

#### 4.6.1 Colour

*Grade 1* — The mango nectar shall be of a good, bright, practically characteristic uniform colour. Any discolouration due to oxidation, processing or other causes shall be considered as a defect.

*Grade 2* — The mango nectar shall be of a reasonably good and of reasonably characteristic uniform colour. The nectar shall be reasonably free from discolouration due to oxidation, processing or other causes.

**4.6.2 Taste and Flavour**

*Grade 1* — The mango nectar shall possess a pleasant aroma and flavour characteristic of the product. The product shall be devoid of any objectionable or off-taste smell or odour.

*Grade 2* — The mango nectar shall possess a pleasant aroma and flavour characteristic of the product. The product shall be devoid of any objectionable or off-taste and reasonably free from smell or odour.

**4.6.3 Absence of Defects**

*Grade 1* — The mango nectar shall be practically free from defects, such as presence of peel or fibrous tissue and other extraneous materials.

*Grade 2* — The mango nectar shall be reasonably free from defects, such as presence of peel or fibrous tissue and other extraneous materials.

**4.7 Total Sugars** — The total sugars (expressed as invert sugar) in the material shall be not less than 15 percent by mass. When tested according to the method prescribed in Appendix B.

NOTE — If agreed to between the purchaser and the vendor, for export purposes only, the total sugars in the material shall be not less than 10 percent by mass.

**4.8** The mango nectar shall also conform to the requirements prescribed in Table 1 and Table 2.

**TABLE 1 REQUIREMENTS FOR MANGO NECTAR**

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. OF IS : 2860-1964*)
(1)	(2)	(3)	(4)
i)	Vacuum of the can, mm, <i>Min</i>	150	5
ii)	Head space of the can, mm, <i>Max</i>	12	6
iii)	Degree Brix of clear sample, <i>Min</i>	12	9
iv)	Acidity (as anhydrous citric acid), percent by mass, <i>Min</i>	0.25	10
v)	Microbiological requirement	To satisfy the requirements of the test	18

\*Method of sampling and test for processed fruits and vegetables.

**TABLE 2 UNITS FOR POISONOUS METALS**

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. OF IS : 2860-1964*)
(1)	(2)	(3)	(4)
i)	Arsenic, mg/kg, <i>Max</i>	0.2	13
ii)	Lead, mg/kg, <i>Max</i>	0.3	14
iii)	Copper, mg/kg, <i>Max</i>	5.0	15
iv)	Zinc, mg/kg, <i>Max</i>	5.0	16
v)	Tin, mg/kg, <i>Max</i>	250	17

\*Methods of sampling and test for processed fruits and vegetables.

## 5. PACKING AND MARKING

### 5.1 Packing

**5.1.1** The material shall be packed in glass bottles or cans of electrolytic or any suitable tinplate. The cans shall be either plain or lacquered internally and hermetically sealed. The side seam shall also be lacquered. The internal lacquer shall be acid-resistant, it shall not be destroyed, altered or its components transferred to the material inside the can during processing or subsequent storage or transport. The can exterior shall be free from dents, rust, perforations and seam distortions. The cans shall not show leaking, panelling, or swell. The interior of the can shall not show any rusting or pitting and the inside lacquer shall be in good condition. The interior of the plain cans may show visible discolouration. Normal feathering shall not be considered as a defect.

**5.1.2** The cans shall be filled with the material, without impairment of quality. The size of the cans and the net mass of their contents shall ordinarily be as given in Table 3. For determining the capacity and dimensions of cans method given in IS : 6093-1971\* shall be followed. In case containers other than those specified in Table 3 are used, the size of the container and the net mass of the contents shall be as agreed to between the purchaser and the vendor.

TABLE 3 SIZES AND CAPACITIES OF CANS

SL No.	TRADE SIZE	NOMINAL DIAMETER	NOMINAL HEIGHT	NET MASS OF CONTENTS
(1)	(2)	(3) mm	(4) mm	(5) g
i)	301 × 409	77.8	115.9	425
ii)	307 × 408	87.3	114.3	540
iii)	401 × 411	103.2	119.1	850
iv)	603 × 700	157.2	177.8	3 200

**5.1.3 Packing in Cases** — The cans shall be packed in wooden packing cases (see IS : 1503-1967†) or corrugated board boxes or any other case as agreed to between the purchaser and the vendor.

**5.2 Marking** — Each container shall be marked with the following particulars:

- Name and grade of the material with the brand name, if any;
- Name and address of the manufacturer;

\*Method for determining the capacity and dimensions of hermetically sealed metal food containers.

†Specification for wooden packing cases (first revision).

**IS : 3547 - 1976**

- c) Net mass of the contents in grams;
- d) Date of manufacture or code number indicating the date of manufacture (to be embossed);
- e) List of permitted additives, if used; and
- f) Manufacturing licence number.

NOTE — Any other markings required under Packaged Commodities Regulations, 1975 shall also be marked.

**5.2.1** Each packing case shall also be marked with the following particulars:

- a) Name of the product,
- b) Name of the manufacturer,
- c) Manufacturing licence number, and
- d) Number of containers  $\times$  mass of each container.

**5.3** The container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## **6. SAMPLING**

**6.1** Representative samples of the material shall be drawn according to the method prescribed in 3 of IS : 2860-1964\*.

## **7. TESTS**

**7.1** The samples of mango nectar shall be tested for ascertaining conformity to the requirements of this specification by the methods referred to in col 4 of Tables 1 and 2, and Appendix B.

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\*Methods of sampling and test for processed fruits and vegetables.

## **APPENDIX A**

*(Clause 4.6)*

### **DETERMINATION OF THE GRADE OF THE PRODUCT**

#### **A-1. APPARATUS**

**A-1.1 White Porcelain Bowls** — big enough to hold the contents of the container under examination.

**A-1.2 Stainless Steel Spoons** — Tablespoons.

#### **A-2. PROCEDURE**

**A-2.1 Panel of Judges** — Grades of the product shall be judged by a panel of three to five judges. All the judges constituting a panel shall be conversant with the factors governing the quality of the product. The containers shall be opened and the contents poured separately into white porcelain bowls. Each judge shall independently examine the contents from each of the containers and indicate scores for different characteristics.

**A-2.1.1** The judges shall consider the following characteristics: a) Colour; b) Taste and flavour; and c) Absence of defects.

**A-2.2 System of Scoring** — The variations within each factor are so described in Table 4 that the scores may be ascertained for each factor and expressed numerically. The relative importance of each factor has been expressed numerically on a scale of 100. Each judge shall indicate the score for the individual factors, by the method described in Table 4 and record his observations in the Score Sheet.

**A-2.2.1** The scores as number of points given by the judges for the contents of each container for the three factors (*see A-2.1.1*) shall be recorded in a tabular form in the Score Card and the average score calculated for each factor with overall average for each container entered in the appropriate column (*see Table 4 and A-2.3.2*).

#### **A-2.3 Ascertaining the Grade**

**A-2.3.1 Consistency Among Judges** — To ascertain the consistency of judgement among the judges, the total score indicated by each of them for the contents of the same container shall be calculated by adding up the scores for the various individual characteristics. If the difference between the maximum and the minimum of the total scores so obtained does not exceed  $(K+5)$ , where  $K$  is the number of judges, the scoring shall be deemed as consistent for the container under consideration. If the difference exceeds

( $K + 5$ ) the score, that is the farthest from its immediate neighbour (the scores being arranged in one order), shall be discarded and the consistency among the remaining judges shall be examined.

**TABLE 4 METHODS FOR INDICATING SCORES FOR MANGO NECTAR**  
(Clauses A-2.2 and A-2.2.1)

SL No.	ORGANOLEPTIC CHARACTERISTIC	DESCRIPTION	MAXIMUM NUMBER OF POINTS (4)
1)	(2)	(3)	
i)	Colour	Good, bright, uniform colour free from discolouration due to oxidation, processing or other causes	25
		Colour not very bright; a very slight variation in the shade of the colour	19
		Colour not very homogeneous; some discolouration	16
		Not very uniform; dull; discolouration	0
ii)	Taste and flavour	Pleasant aroma, flavour characteristic of the mango nectar; free from objectionable, off-taste smell and odour	50
		Flavour not strong, slightly metallic taste	37
		Taste of slightly immature mangoes, some offensive smell	33
		Flat taste, offensive odour. No flavour	0
iii)	Absence of defects	Free from defects, no peel, fibrous tissue or other extraneous material present	25
		Presence of some fibres, very slight grit present	19
		Rather fibrous, a piece of peel or other thickening; gritty grains easily felt	16
		Wholly fibrous; peel or other substances present	0

**A-2.3.2** When the consistency (*see* A-2.3.1) is thus established, the overall average scores indicated by the judges whose scoring has been found to be consistent shall be calculated for each container. The average score for each of the individual characteristics shall also be calculated by taking into account the corresponding scores as given by the same judges for the contents of the same container.

**A-2.3.3 Assignment of Grade** — In order to assign a grade for the contents of a container, the following procedure shall be adopted:

**Grade 1** — The score for each factor individually shall be not less than 75 percent of the maximum score obtainable and the overall average score shall be not less than 85 points.

**Grade 2** — The score for each factor individually shall be not less than 65 percent of the maximum score obtainable, and the overall average score shall be not less than 75 points.

# **SCORE SHEET FOR INDIVIDUAL JUDGE**

Sample No. ....

Date of Sampling.....

*Details of the Sample:*

- a) Product ..... b) Name of Manufacturer ..... c) Type. ....  
d) Batch No..... e) Date of Manufacture.....

FACTORS	SCORE POINTS	SAMPLE CANS									
		1	2	3	4	5	6	7	8	9	10
Colour	Grade 1 : 19 - 25										
	Grade 2 : 16 - 18										
Taste and Flavour	Grade 1 : 37 - 50										
	Grade 2 : 33 - 37										
Absence of Defects	Grade 1 : 19 - 25										
	Grade 2 : 16 - 18										

Signature of the Judge .....

Date .....

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**SCORE CARD**

Sample No. ....

Date of Sampling.....

*Details of the Sample Can :*

a) Product ..... b) Name of Manufacturer..... c) Type .....

d) Batch No. .... e) Date of Manufacture .....

FACTOR	COLOUR	TASTE AND FLAVOUR	ABSENCE OF DEFECTS	TOTAL SCORES	AVERAGE SCORE FOR				GRADE OF THE CAN
JUDGE	A B C D E	A B C D E	A B C D E	A B C D E	COLOUR AND TEXTURE	TASTE AND FLAVOUR	ABSENCE OF DEFECTS	TOTAL	
CAN NUMBER									

**APPENDIX B****( Clause 4.7 )****DETERMINATION OF TOTAL SUGARS****B-1. METHOD****B-1.1 Reagents****B-1.1.1 Sodium Hydroxide Solution** — Approximately 0.1 N.

**B-1.1.2 Stock Solution of Invert Sugar** — Weigh accurately 9.5 g of pure sucrose and transfer it to a 1-litre volumetric flask with 100 ml of water. Add 5 ml of concentrated hydrochloric acid. Allow this to stand for 3 days at 20 to 25° C, and then make up to volume with water. This solution is stable for several months.

**B-1.1.3 Standard Solution of Invert Sugar** — Neutralize a known aliquot of the stock solution of invert sugar. (see **B-1.1.2**) with sodium hydroxide solution using litmus paper. Dilute with water to a known volume, so that more than 15 ml but less than 30 ml of it shall be required to reduce all the copper in Fehling's solution taken for titration. Note the concentration of invert sugar in this solution as mg per 100 ml (see Note). Prepare this solution fresh everyday.

NOTE — When 10 ml of Fehling's solution is taken for titration, a standard invert sugar solution containing 0.12 to 0.30 percent (m/v) of invert sugar is used.

**B-1.1.4 Methylene Blue Indicator Solution** — Dissolve 0.2 g of methylene blue in water and dilute to 100 ml.

**B-1.1.5 Fehling's Solution (Soxhlet Modification)** — Prepared by mixing immediately before use, equal volumes of Solution A and Solution B, prepared as given below:

**Solution A** — Dissolve 34.639 g of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in water, add 0.5 ml of concentrated sulphuric acid of r.d. 1.84 and dilute to 500 ml in a volumetric flask. Filter the solution through prepared asbestos.

**Solution B** — Dissolve 73 g of Rochelle salt (potassium sodium tartarate —  $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ ) and 50 g of sodium hydroxide in water, dilute to 500 ml in a volumetric flask and allow the solution to stand for 2 days. Filter this solution through prepared asbestos.

**B-1.1.5.1 Standardization of Fehling's solution** — Pour standard invert sugar solution (see **B-1.1.3**) into a 50-ml burette (see Notes under **B-1.2.3**). Pipette 10 ml of Fehling's solution into a 300-ml flask and run in

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from the burette almost the whole of the standard invert sugar solution required to effect reduction of all the copper, so that not more than 1 ml will be required later to complete the titration. Heat the flask containing the mixture over a wire gauze. Boil the contents of the flask gently for 2 minutes. At the end of a 2 minutes of boiling, add without interrupting boiling, 1 ml of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add standard invert sugar solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears. [The titration should be completed within 1 minute, so that the contents of the flask boil altogether for 3 minutes without interruption (see Note 2 under B-1.2.3)]. Note the titre, that is, the total volume in millilitres of standard invert sugar solution used for the reduction of all the copper in 10 ml of Fehling's solution.

**B-1.1.6 Zinc Acetate Solution** — Dissolve 21.9 g of crystallized zinc acetate [ $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ ] in water and add 3 ml of glacial acetic acid. Make up to 100 ml.

**B-1.1.7 Potassium Ferrocyanide Solution** — Dissolve 10.6 g of crystalline potassium ferrocyanide and make up to 100 ml with water.

**B-1.1.8 Concentrated Hydrochloric Acid** — r.d. 1.16.

**B-1.1.9 Concentrated Ammonia Solution** — r.d. 0.88.

**B-1.1.10 Dilute Ammonia Solution** — 10 ml of concentrated ammonia solution diluted to 100 ml with water.

**B-1.1.11 Dilute Acetic Acid Solution** — approximately equivalent to the dilute ammonia solution in strength.

## **B-1.2 Procedure**

**B-1.2.1 Preparation of the Solution** — Weigh accurately about 40 g of the well mixed sample and transfer to a 100 ml beaker. Add 50 ml of hot water at 80 to 90°C. Mix and transfer to a 200 ml measuring flask, washing it with successive quantities of distilled water at 60°C, until the volume is 120 to 150 ml. Mix and cool to room temperature and add 5 ml of dilute ammonia solution. Mix and allow to stand for 15 minutes. Add the exact equivalent of dilute acetic acid to neutralize the ammonia added. Mix and add 12.5 ml of zinc acetate solution followed by 12.5 ml of potassium ferrocyanide solution. Mix again. Make up to 200 ml mark. Allow to settle and filter. Mark this solution X.

Pipette 50 ml of solution X into a 100-ml volumetric flask, add 5 ml of concentrated hydrochloric acid and heat at 60°C for 5 minutes. Cool the

solution and neutralize with sodium hydroxide solution. Mark this solution  $Y$ . Make up to 100 ml.

Dilute the solutions  $X$  and  $Y$  so that the volume of solution required to react with 10 ml Fehling's solution is between 15 and 50 ml (see B-1.2.2). Mark them  $X_1$  and  $Y_1$  respectively.

**B-1.2.2 Incremental Method of Titration** — Pour the prepared solution  $Y$  (see B-1.2.1) into a 50-ml burette (see Note 3 under B-1.2.3). Pipette 10 ml of Fehling's solution into a 300-ml conical flask and run in from the burette 15 ml of the solution. Without further dilution heat the contents of the flask over a wire gauze, and boil. (After the liquid has boiled for about 15 seconds, it will be possible to judge by the bright red colour imparted to the boiling liquid by the suspended cuprous oxide, if the copper is almost all reduced.) When it is judged that nearly all the copper is reduced, add 1 ml of methylene blue indicator solution (see Note 1 below). Continue boiling the contents of the flask for 1 to 2 minutes from the commencement of ebullition, and then add the prepared solution in small quantities (1 ml or less at a time), allowing the liquid to boil for about 10 seconds between successive additions, till the blue colour of the indicator just disappears (see Note 2 under B-1.2.3). After the mixture of Fehling's solution with 15 ml of the prepared solution has been boiling for a quarter of a minute there appears to be still much unreduced copper, add the prepared solution from the burette in large increments (more than 1 ml at a time, according to judgement), and allow the mixture to boil for a quarter of a minute after each addition. Repeat the addition of the prepared solution at intervals of 15 seconds until it is considered unsafe to add a large increment of the prepared solution. At this stage, continue to boil for an additional 1 to 2 minutes; add 1 ml of methylene blue indicator solution and complete titration by adding the prepared solution in small quantities (less than 1 ml at a time) (see Note 2).

**NOTE 1** — It is advisable not to add the indicator until the neighbourhood of the end point has been reached, because the indicator retains its full colour until the end point is almost reached and thus gives no warning to the operator to go slowly.

**NOTE 2** — When the operator has had a fair amount of experience with the method a sufficiently accurate result may often be obtained by a single estimation by the incremental method of titration, but for utmost degree of accuracy, a second titration should be carried out by the standard method of titration (see B-1.2.3).

**B-1.2.3 Standard Method of Titration** — Pipette 10 ml of Fehling's solution into a 300-ml conical flask and run in from the burette almost the whole of the prepared solution  $Y_1$  required to effect reduction of all the copper (determined under B-1.2.2), so that, if possible, not more than 1 ml shall be required later to complete the titration. Boil the contents of the flask gently for 2 minutes. At the end of a 2 minutes of boiling, add without interrupting boiling, 1 ml of methylene blue indicator solution.

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While the contents of the flask continue to boil, begin adding the prepared solution (one or two drops at a time), from the burette till the blue colour of the indicator just disappears (*see Note 1*). The titration should be completed within 1 minute, so that the contents of the flask boil altogether for 3 minutes without interruption.

**NOTE 1** — The indicator is so sensitive that it is possible to determine the end point within one drop of the prepared solution in many cases. The complete decolourization of the methylene blue is usually indicated by the whole reaction liquid in which cuprous oxide is continuously churned up becoming bright red or orange in colour. In case of doubt, the flame may be removed from the wire gauze for 1 or 2 seconds and the flask held against a sheet of white paper. (A holder of paper, suitably fixed round the neck of the flask is very convenient for this purpose as it can be left round the neck of the flask without risk of overbalancing it.) The top edge of the liquid would appear bluish if the indicator is not completely decolourized. It is not advisable to interrupt the boiling for more than a few seconds as the indicator undergoes back oxidation rather rapidly when air is allowed free access into the flask, but there is no such danger as long as a continuous stream of steam issues from the mouth of the flask.

**NOTE 2** — It should be observed that with both incremental and standard methods of titration, the flask containing the reaction mixture is left on the wire gauze over the flame throughout the titration, except when it may be removed for a few seconds to ascertain if the end point is reached.

**NOTE 3** — In adding sugar solution to the reaction mixture the burette may be held in hand over the flask. The burette may be fitted with a small outlet tube bent twice at right-angles, so that body of the burette can be kept out of the stream while adding sugar solution. Burette with glass taps are unsuitable for this work, as the taps become heated by the steam and are liable to jam.

**B-1.2.4** Repeat the titration as given in **B-1.2.2** and **B-1.2.3** using solution  $X_1$  (*see B-1.2.1*).

## **B-2. CALCULATION**

**B-2.1** Total sugars (as invert sugar),  
percent by mass 
$$= \frac{20\ m}{M} \left[ \frac{f_2}{v_2} - \frac{f_1}{v_1} \right]$$

where

- $m$  = mass in mg of sucrose corresponding to 10 ml Fehling's solution (*see B-1.1.5.1*);
- $M$  = mass in g of the material taken for the determination (*see B-1.2.1*);
- $f_2$  = dilution factor for solution  $X_1$  from solution  $X$  (*see B-1.2.1*);
- $v_2$  = volume in ml of solution  $X$  corresponding to 10 ml Fehling's solution (*see B-1.2.4*);
- $f_1$  = dilution factor for solution  $Y_1$  from solution  $Y$  (*see B-1.2.1*); and
- $v_1$  = volume in ml of solution  $Y_1$  corresponding to 10 ml Fehling's solution (*see B-1.2.3*).

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